

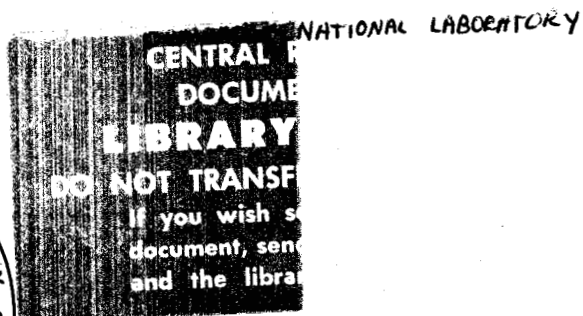
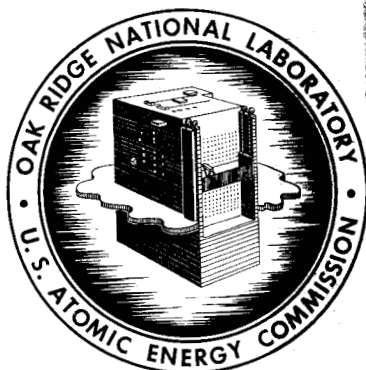
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FURTHER EVALUATION OF AMINES AS
EXTRACTANTS FOR URANIUM FROM
SULFATE LIQUORS

W. D. Arnold
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OAK RIDGE NATIONAL LABORATORY
operated by
UNION CARBIDE CORPORATION
for the
U.S. ATOMIC ENERGY COMMISSION

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FOR URANIUM FROM SULFATE LIQUORS

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ABSTRACT

Approximately 60 organonitrogen compounds were tested for their ability to extract uranium from acidic sulfate solution. Those showing significant extraction power were tested with respect to other pertinent extraction characteristics, including solubility loss to aqueous liquors, selectivity for uranium, and compatibility with molybdenum liquors. As in previous studies, the simple amines were most useful, a number of the primary, secondary, and tertiary amines showing promise as practicable process extractants.

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1.0 INTRODUCTION

The investigation and evaluation of organonitrogen compounds as extractants for the recovery of uranium, thorium, and other metals of interest to the atomic energy program have been conducted at this laboratory since 1952. Before being considered for process studies, new reagents are subjected to screening tests, these tests being designed to eliminate the less promising compounds in a stepwise manner. Each reagent is first tested for its ability to extract uranium from acidic sulfate solutions. Compounds showing appreciable extraction power are tested for other important properties such as diluent compatibility, solubility loss to the aqueous phase, and selectivity for uranium. Reagents showing the greatest promise in the screening tests are examined in the process development program.

Results from screening studies on over 200 different compounds have been reported previously.¹⁻³ This report summarizes results of screening tests since that time, including tests reported in progress reports for February-December, 1957.

The authors acknowledge the contributions of John G. Moore and Janet Debnam who performed many of the evaluation tests.

2.0 PRELIMINARY URANIUM EXTRACTION TESTS

The ability of each amine to extract uranium was tested by contacting 0.1 M solutions of the amine in various diluents with an acidic sulfate solution (1 g of uranium per liter, 1 M SO_4 , pH 1) and determining the uranium distribution between the phases. Results of these tests are listed in Table 2.1. The table is long and, in order to simplify presentation, observations and conclusions drawn from these data are presented below in itemized fashion. Information on structure and source of supply of each compound is listed in the Appendix (Sec. 7.0).

2.1 Primary Amines

In agreement with previous¹ results, branched-chain primary amines gave good uranium extraction and phase separation. Compounds tested included a new batch of 1-(3-ethylpentyl)-4-ethyloctylamine (21F81) and two new amines, 1-heptyloctylamine and 1-undecyldodecylamine.

Two straight-chain, slightly unsaturated primary amines (Armeen O and Armeen OD), derived from oleic acid, were poor extractants. A series of alkyl ether amines could not be used since they formed permanent emulsions.

Table 2.1. Preliminary Uranium Extraction Tests from Sulfate Solutions with Organonitrogen Compounds

Organic: 0.1 M amine

Aqueous: 1.0 g of uranium per liter, 1 M SO₄, pH 1.0

Phase ratio, a/o: 1/1

Compound	Compound No.	Uranium Extraction Coefficient (E _g)				Phase Separation
		Kerosene	Kerosene + 3 vol % TDA	Chloro-form	Benzene	
A. Primary Amines						
21F81	120C	25	-	65	30	Good
1-Heptyloctyl	270A	35	25	65	30	Good
1-Undecyldodecyl	272A	35	-	100	45	Good
Armeen O	275A	0.5	-	2	0.2	Good
Armeen OD	276A	0.2	-	1	0.2	Good
Alkyl ether amine No. 1	304A	Emulsion	-	Emulsion	Emulsion	-
Alkyl ether amine No. 3	305A	Emulsion	-	Emulsion	Emulsion	-
Alkyl ether amine No. 4	306A	Emulsion	-	Emulsion	Emulsion	-
B. Secondary Amines						
S-24	30D	100	-	1.5	25	Good
Di(1-heptyloctyl)	332A	100	-	-	30	Good
Di(1-nonyldecyl)	341A	-	-	-	40	Good
N-(1-nonyldecyl)-dodecyl	309A	120	-	20	115	Good
N-(1-undecyldodecyl)-dodecyl	308A	125	-	20	90	Good
Di(2-propyl-4-methyl-pentyl)	311A	110	-	90	130	Good
Di(tridecyl)	227B	60	75	-	210	Good
Amberlite LA-1	193G	70	-	3	55	Good
(formerly Amine 9D-178)	193H	70	-	-	60	Good
Amberlite LA-2	325A	10	-	-	35	Poor in
	325B	10	10	-	25	kerosene

Table 2.1 (Cont'd.)

Compound	Compound No.	Uranium Extraction Coefficient (Eq)			Benzene	Phase Separation
		Kerosene	Kerosene + 3 vol % TDA	Chloro-form		
Mixed (n-octyl and n-decyl) secondary	298A	3rd phase	19	130	12	Poor in benzene
N-Benzylisopropyl	307A	Nil	-	Nil	Nil	Good
N-Benzyl-1-(3-ethyl-pentyl)-4-ethyloctyl	228B	>5000	-	70	2000	Good
N-Benzyl-1-isobutyl-3,5-dimethylhexyl	244B	1400	-	150	3000	Good
N-Benzyl(1-nonyldecyl)	330A	2000	2000	-	95	Good
N-Benzyl(1-undecyl-dodecyl)	331A	4000	2000	-	510	Poor in kerosene
N-Benzyl-octadecyl	327A	-	11 ^a	-	b	Good
N-Benzyl(1-methyl-octadecyl)	329A	0.8	2.4 ^c	-	2000	Poor in kerosene
N-(p-sec-amylbenzyl)-1-isobutyl-3,5-dimethylhexyl	251B	140	-	-	90	Good
4-RKB-21, N-(cyclohexylmethyl)-1-isobutyl-3,5-dimethylhexyl	297A	85	-	5	90	Good
4-RKB-20, N-(1-methyl-cyclohexylmethyl)-1-isobutyl-3,5-dimethylhexyl	296A	20	-	2	25	Good
4-RKB-16, N-(2,4,6-trimethylcyclohexylmethyl)-1-isobutyl-3,5-dimethylhexyl	293A	70	-	1.5	25	Good
4-RKB-19, N-cyclohexylmethyl-1-(3-ethyl-pentyl)-4-ethyloctyl	295A	25	-	3	95	Good

Table 2 (Cont'd.)

Compound	Compound No.	Uranium Extraction Coefficient (Eq)				Phase Separation
		Kerosene	Kerosene + 3 vol % TDA	Chloro- form	Benzene	
4-RKB-18, N-(1-methylcyclohexyl-methyl)-1-(3-ethylpentyl)-4-ethyloctyl	294A	120	-	1	35	Good
4-RKB-15, N-(2,4,6-trimethylcyclohexyl-methyl)-1-(3-ethylpentyl)-4-ethyloctyl	292A	130	-	0.8	25	Good
Secondary Rosin Amine X-8523-83-7	277A	3	-	8	15	Good
Rosin Amine HX-N-RA-60A	333A	Ppt	-	-	305	Good in benzene
Rosin Amine HX-N-RA-61A	334A	Insol	20 ^d	-	180 ^e	Good in benzene
Polyrad 0100	282A	Ppt	-	Ppt	0.7	Poor
C. Tertiary Amines						
Alamine 336	299C	80	-	-	110	Good
ADM Mixed Tertiary	328A	140	200	-	140	Poor in kerosene
Dilauryl-n-butyl	101B	55	100	-	180	Poor in kerosene
Trilauryl	86B	80	80	5	150	Good
Armeen 3-12 (trilauryl)	86C	65	75	5	145	Poor
Trilauryl	86D	160	-	-	185	Good
Di(2-ethylhexyl)-n-hexyl	195A	0.4	-	0.6	1	Good
Tri(iso-heptyl)	326A	3rd phase	95	-	85	Good
Tri(iso-octyl)	239A	-	100 ^f	0.6	100	Good
	239B	-	80	0.8	95	Good
	239C	-	60	1	60	Good

Table 2.1 (Cont'd.)

Compound	Compound No.	Uranium Extraction Coefficient (E_{U})				Phase Separation
		Kerosene	Kerosene + 3 vol % TDA	Chloro-form	Benzene	
Tris(tridecyl)	310A	145	30	0.2	65	Good
Amberlite XE-204	253A	-	70	3	130	Good
	253B	-	60	3	140	Good
	253C	-	90	-	240	Good
Armeen M-2S	249A	7	-	55	75	Poor except in CHCl_3
Armeen M-2HT	250A	0.2	-	40	60	Poor except in CHCl_3
Propomeen 212/11	313A	2	-	-	16	Very poor
Propomeen 212/12	319A	2	-	-	20	Poor
Propomeen 212/13	320A	2	-	-	15	Poor
Propomeen 212/14	322A	4	-	-	25	Poor
Propomeen 212/20	323A	4	-	-	20	Poor
Propomeen 212/50	324A	15	-	-	25	Good
RD-2805B	300A	Nil	-	-	Nil	Poor
RD-2806B	301A	Nil	-	-	Nil	Good
RD-2807B	302A	Nil	-	-	3rd phase	Good
RD-2808B	303A	3rd phase	-	-	12	Poor
Polyrad 0200	283A	Emulsion	-	1	3rd phase	Poor
D. Miscellaneous Nitrogen Compounds						
Arquad 18	291A	Insol	-	-	Insol	-
Quaternary B-104 ^g	247A	0.6	-	0.7	0.8	Good
Arquad 2C	105B	Ppt	-	-	0.6	Poor
Arquad 2T	290A	Insol	-	1	0.6	Emulsions
N,N'-bis-(1-heptyl-octyl)ethylenediamine	271A	3rd phase	-	2	3rd phase	Poor
Diamine 26	281A	Ppt	-	0.6	Ppt	Poor
Di(1-aminocyclohexyl-methyl)amine	312A	Nil	-	Nil	Nil	Poor in kerosene
Fatchemco DM-O ^h	256A	Nil	-	-	Nil	Emulsions

Table 2.1 (Cont'd.)

Compound	Compound No.	Uranium Extraction Coefficient (E_a^O)				Phase Separation
		Kerosene	Kerosene + 3 vol % TDA	Chloro-form	Benzene	
Deriphat 150A	278A	Insol	-	Insol	Insol	-
Arneel Si ⁱ	269A	Nil	-	Nil	Nil	Good
Alkylaniline C-5 ^h	254A	Nil	-	0.1	3rd phase	Good
Ethoquad C/25 ^j	314A	Insol	-	-	Insol	-
O. C. No. 30 ^j	315A	Insol	-	-	Insol	-
Emery 578-42-R	288A	Emulsion	-	Emulsion	Emulsion	-
Duomeen T salt of polydodecyl-benzene-sulfonic acid ⁱ	284A	Nil	-	Nil	Nil	Poor in kerosene
Aminopolybutene ⁱ	285A	Nil	-	Nil	Nil	Good
Imidazoline from fatty acid mixture and triethylenetetramine	286A	Nil	-	-	Ppt	Poor

^a0.05 M amine in kerosene + 10 vol % TDA.

^bIn benzene + 5 vol % TDA.

^cIn kerosene + 5 vol % TDA, $E_a^O = 70$.

^dIn kerosene + 5 vol % TDA.

^eExtract formed a gel upon standing.

^f2 vol % capryl alcohol added to kerosene diluent.

^gContained some isopropanol since the compound was added as 65% solution in isopropanol.

^h0.05 M amine.

ⁱ10 vol % amine.

^jAmine concentration = 50 g/liter.

2.2 Secondary Amines

Performances of the branched-chain secondary amines were similar to those observed previously^{1,2} with other compounds of this type. A new batch of S-24 and two new amines, di(1-heptyloctyl) and di(1-nonyldecyl), with symmetrical alkyl chains joined to the nitrogen through a secondary carbon showed extraction coefficients of ~100 in kerosene and ~30 in benzene diluent. In contrast, with chloroform as the diluent the coefficient for S-24 was only 1.5. With compounds containing one straight alkyl chain and one alkyl chain branched close to the nitrogen, e.g., 1-(nonyldecyl)dodecylamine, coefficients in kerosene were about the same and the coefficients in benzene and chloroform much higher than for S-24. Amines with branching farther from the nitrogen, di(2-propyl-4-methylpentyl)amine and di(tridecyl)amine, gave higher coefficients in benzene (130-200) than in kerosene (60-110), whereas Amberlite LA-1, in which both chains are highly branched and one is unsaturated, gave about the same coefficients (60-70) in the two diluents. Results with two batches of the latter compound were in agreement with those for previous² batches. Amberlite LA-2, which has one highly branched and one straight chain, was a much weaker uranium extractant than Amberlite LA-1. Phase separation of the former, which was slow in kerosene, was satisfactorily rapid in kerosene modified with 3 vol % tridecanol.

Coefficients for a straight chain (mixed *n*-octyl and *n*-decyl) secondary amine were relatively low (10-20) in kerosene-alcohol or benzene but high (130) in chloroform diluent.

The extremely high extraction coefficients (>1000) obtained previously² with N-benzyl-1-(3-ethylpentyl)-4-ethyloctylamine were also obtained with a new sample of this compound. Results of tests with several other N-benzyl alkyl secondary amines showed that the structure of the alkyl chain and the choice of diluent drastically affected uranium extractions. With a short alkyl chain (N-benzylisopropylamine), extraction of uranium was negligible, probably owing to excessive loss of amine to the aqueous phase. Branching on the carbon adjacent to the nitrogen was beneficial, three compounds of this type, i.e., N-benzyl-1-isobutyl-3,5-dimethylhexylamine, N-benzyl-1-nonyldecylamine, and N-benzyl-1-undecyldodecylamine, having extraction coefficients in kerosene approximately equivalent to the structurally similar N-benzyl-1-(3-ethylpentyl)-4-ethyloctylamine. N-Benzyl-octadecylamine was insoluble in kerosene. Modification with 10 vol % TDA was necessary to keep 0.05 *M* amine in solution and the extraction coefficient was only 11. In benzene, 5 vol % TDA was needed to prevent third phase formation and the extraction coefficient (0.1 *M* amine) was 200. Adding a small amount of branching to this compound, i.e., N-benzyl-(1-

methyloctadecyl)amine, improved diluent compatibility and raised the coefficient in benzene to 2000, but the coefficient in kerosene was <1. Substitution of an alkyl group in the benzene ring greatly impaired extractions, i.e., N-(p-sec amylbenzyl)-1-isobutyl-3,5-dimethylhexylamine ($E_a^O = 140$) vs. N-benzyl-1-isobutyl-3,5-dimethylhexylamine ($E_a^O = 1400$).

A series of cyclohexylmethyl compounds showed good extraction in benzene and kerosene and poor extraction in chloroform, typical behavior for highly branched secondary amines. None of these compounds showed unusually high extraction coefficients, indicating that the benzene ring in the benzyl-substituted compounds (above) makes an important contribution to their high extraction power.

Three secondary amines derived from rosin gave good extractions in benzene but had poor compatibility with kerosene. A related amine also carrying hydroxyl groups (Polyrad 0100) gave negligible extraction.

2.3 Tertiary Amines

As in previous studies,¹⁻² long-chain tertiary amines with straight chains or with branching distant from the nitrogen gave good extractions in kerosene, kerosene-alcohol, and benzene but much weaker extractions in chloroform. Compounds of this type tested included Alamine 336 (mixed n-octyl and n-decyl), ADM mixed tertiary (dilauryl-n-decyl and dilauryl-n-octyl), dilauryl-n-butyl, trilauryl, tri(iso-heptyl), tri(iso-octyl), tris(tridecyl), and Amberlite XE-204 (didodecenyl-n-butyl) amines. Certain of the amines, including XE-204, dilauryl-n-butyl and tri(iso-heptyl), required addition of alcohol to the kerosene diluent to avoid slow-breaking emulsions or third phase formation.

Armeen M-2S, a methyl dialkyl amine derived from soya, and Armeen M-2HT, a similar compound derived from hydrogenated tallow, showed moderate extractions in kerosene and good extractions in benzene and chloroform. Phase separation with these compounds was slow except in chloroform.

Extraction coefficients for a tertiary amine with branching close to the nitrogen, di(2-ethylhexyl)-n-hexylamine, were low (0.4-1), probably due to steric hindrance.

A series of dilauryl (polypropoxy) amines (Propomeens) with 1, 2, 3, 4, 10, and 40 propoxy units in a polyether chain showed only fair extraction, lower in kerosene than in benzene. The highest weight amine showed less difference than the others between benzene and kerosene, and was the only one which did not form emulsions in kerosene. A series of alkyl (di-isopropoxy) amines were poor extractants.

2.4 Miscellaneous Organonitrogen Compounds

No significant extraction power ($E_a^0 < 1$) was shown by any of 17 compounds examined, including six quaternary ammonium compounds.*

3.0 SOLUBILITY LOSS OF AMINES TO AQUEOUS SOLUTIONS

For economy, the distribution loss of amine to the aqueous waste streams must be low, particularly when treating liquors very dilute in uranium. Solubility loss measurements for amines which extracted uranium effectively in preliminary tests (Sec. 2.0) showed that, for most of these compounds, loss of amine by this mechanism was sufficiently low to be of only minor economic importance.

The test procedure, described in detail in previous reports,¹⁻² consisted of equilibration of the amine (dissolved in kerosene or kerosene-alcohol) with a synthetic leach liquor over a range of aqueous/organic phase ratios up to 100/1 or higher, reconverting the amine salt to free amine by contact with excess base, and determining the residual amine concentration in the solvent phase. Plotting the final amine concentration against the phase ratio usually gave a curve with a relatively steep initial slope, which changed rather abruptly to a much lower constant slope (Fig. 3.1). The high loss of amine to the first few aqueous volumes contacted is attributed to the loss of relatively water-soluble titratable bases (probably low-molecular-weight amines) present as impurities. This loss (termed "initial loss") is expressed as percent of original amine and, from the process viewpoint, would be reflected only as a moderate increase in the purchase price of the amine. The loss rate defined by the linear portion of the curve is that of the principal amine and is termed the "steady-state" loss. In most cases the loss rate was measured to synthetic uranium-barren Marysville leach liquor, but some of the primary amines were tested with synthetic Blind River liquors since these compounds have shown utility for recovering thorium from this source.

Two of the primary amines, 1-nonyldecyl and 1-undecyl-dodecyl, showed negligible (<5 ppm) steady-state losses,

*Significant extraction of uranium from sulfate solutions has been obtained previously, however, with other quaternary ammonium compounds.¹ In addition, the quaternaries are effective extractants for uranyl carbonate²⁻⁴ and strong extractants for some metal nitrates.⁵ One of the more useful compounds, Aliquat 336 (General Mills), is now commercially available.

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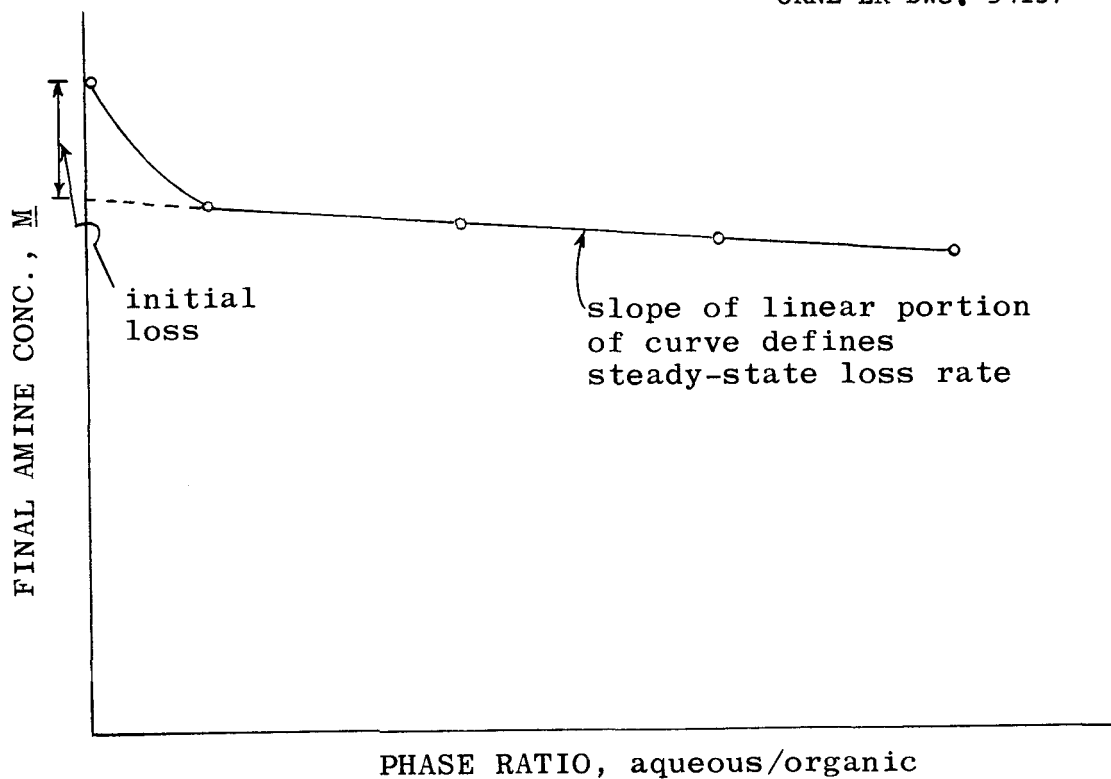


Fig. 3.1 Typical amine solubility loss curve.

whereas the losses of 21F81 and 1-heptyloctylamine were ~20 ppm. As with previous² batches, the initial loss of Primene JM-T (a homologous mixture of primary amines) was high (33%). The loss rate for the scrubbed amine did not reach a steady-state value but was still decreasing even after contact with 2000 aqueous volumes:

Range of Phase Ratios (a/o)	Cumulative Loss, % of initial ^a amine	Average Loss Rate, ^b ppm
0-500	27	19
500-1000	41	10
1000-1500	48	5
1500-2000	54	4

^aRefers to amine concentration after scrubbing (~0.1 M)

^bTo synthetic Blind River liquor; analysis in Table 3.1.

Table 3.1. Solubility Loss of Amines to Aqueous Solutions

Organic: 0.10 M amine in kerosene with indicated concentration of tridecanol (TDA)

Aqueous: A - Synthetic uranium-barren Marysvale leach liquor containing 5.8 g of Fe(III), 3.3 g of Al, 1.7 g of F, 2 g of PO₄ and 50 g of SO₄ per liter, pH 0.9

B - Synthetic uranium- and thorium-barren Blind River liquor containing 1.0 g of Fe(II), 1.0 g of Fe(III), 1.5 g of Al, and 20 g of SO₄ per liter, pH 1.4

C - Synthetic Blind River ion exchange effluent containing 2.4 g of Fe(II), 0.5 g of Fe(III), 0.9 g of Al, 0.03 g of Ti, 0.4 g of Cl, 1.0 g of NO₃, and 15 g of SO₄ per liter, pH 1.8

Procedure: Organic and aqueous equilibrated at a number of different phase ratios; amine converted to free amine by contact with excess base and titrated with 0.05 M HClO₄ in dioxane

Compound	Comp'd No.	TDA Conc., vol %	Aqueous	Amine Loss		Maximum Phase Ratio (a/o)
				Initial, %	Steady-State, ppm ^a	
<u>Primary Amines</u>						
21F81	120C	0	A	0	17	125
1-Heptyloctyl	270A	3	A	0	20	100
1-Nonyldecyl ^b	336A	2	B	<1	<5	200
1-Nonyldecyl ^b	336B	2	B	<1	<5	400
1-Undecyldodecyl	272A	3	A	8	<5	100
1-Undecyldodecyl ^b	272B	2	B	<1	<5	200
Primene JM-T	6E	0	C	33 ^c	~10 ^d	2000
<u>Secondary Amines</u>						
S-24	30B	0	A	2	9 ^e	1800
Di(1-heptyloctyl)	332A	0	A	1	<5	400
Di(1-nonyldecyl)	341A	0	A	<1	<5	400
N-(1-nonyldecyl)-dodecyl	309A	0	A	<1	<5	500
N-(1-undecyl-dodecyl)dodecyl	308A	0	A	11	<5	500
Di(2-propyl-4-methylpentyl)	311A	0	A	-	>270	500
Di(tridecyl)	227B	3	A	2	<5	500
Amberlite LA-1	193H	0	A	6	14 ^d	1500
Amberlite LA-2	325A	0	A	<1	<5	500
	325B	0	A	<1	<5	500
Mixed (n-octyl and n-decyl) secondary	298A	5	A	13	41	100

Table 3.1 (Cont'd.)

Compound	Comp'd No.	TDA Conc., vol % Aqueous		Amine Loss		Maximum Phase Ratio (a/o)
				Initial, %	Steady-State, ppm ^a	
N-Benzyl-1-(3-ethylpentyl)-4-ethyloctyl	228B	0	A	<1	<5	500
N-Benzyl-(1-nonyldecyl)	330A	3	A	2	<5	500
N-Benzyl-(1-undecyldodecyl)	311A	3	A	<1	<5	500
N-Benzyl-octadecyl	327A	10	A	<1	<5	500
N-Benzyl-(1-methyloctadecyl)	329A	5	A	f	f	-
N-(p-sec-amyl-benzyl)-1-iso-butyl-3,5-dimethylhexyl	251B	0	A	8	13	100
4-RKB-21	297A	0	A	-	>500	200
4-RKB-20	296A	0	A	-	>500	200
4-RKB-16	293A	0	A	15	100	200
4-RKB-19	295A	0	A	<1	5	200
4-RKB-18	294A	0	A	<1	<5	500
4-RKB-15	292A	0	A	<1	<5	200
<u>Tertiary Amines</u>						
Alamine 336	299A	3	A	<1	<5	500
	299B	3	A	1	<5	500
	299C	3	A	2	<5	500
	299D	3	A	2	<5	400
ADM Mixed Tertiary	328A	0	A	1	<5	500
Dilauryl-n-butyl	101B	0	A	9	<5	500
Trilauryl	86B	0	A	<1	<5	200
	86C	3	A	<3	<5	500
	86D	0	A	<1	<5	500
Tri(iso-heptyl)	326A	3	A	g	g	500
Tri(iso-octyl)	239C	3	A	4	~11 ^d	1400
Tris(tridecyl)	310A	0	A	<1	<5	500
Amberlite XE-204	253C	3	A	3	<5	400
Armeen M-2C	248A	3	A	3	13	100

^aParts of amine per million parts of aqueous.

^bInitial amine concentration was 0.05 M.

^cInitial amine concentration was 0.15 M; scrubbed with 30 volumes of 0.2 M H₂SO₄ which reduced amine concentration to 0.1 M (33% loss); scrubbed amine used for steady-state loss test; see text for additional data.

Table 3.1 (Cont'd.)

- ^dNot a true steady-state loss since loss curve was not linear; see text.
- ^eLoss curve not linear until after contact with several hundred aqueous volumes; see text for additional data.
- ^fFormed permanent emulsions.
- ^g70% of the initial amine was lost to the first 100 aqueous volumes and 90% to 200 volumes.

Excluding the initial loss, the average loss of amine to the 2000 aqueous volumes was ~10 ppm, a loss rate much lower than that estimated previously² at lower aqueous/organic phase ratios to solutions of different composition. This lower loss rate would allow application of Primene JM to the recovery of thorium from liquors more dilute in thorium than had previously been considered economically practicable.

A number of secondary amines including di(1-heptyloctyl), di(1-nonyldecyl), N-(1-nonyldecyl)dodecyl, N-(1-undecyldodecyl)-dodecyl, di(tridecyl), Amberlite LA-2, N-benzyl-1-(3-ethyl-pentyl)-4-ethyloctyl, N-benzyl(1-nonyldecyl), N-benzyl(1-undecyldodecyl), N-benzyl-octadecyl, 4-RKB-18, and 4-RKB-15 showed negligible (<5 ppm) steady-state losses. In contrast, losses of di(2-propyl-4-methylpentyl), 4-RKB-21, 4-RKB-20, and 4-RKB-16 amines were too high for these compounds to be of practicable interest. As with Primene JM, the loss rate curve for Amberlite LA-1 (a homologous mixture of amines) did not become linear even after contact with 1500 aqueous volumes. The average loss to the 1500 aqueous volumes, excluding the 6% initial loss to the first 30 volumes, was ~14 ppm. The curve for S-24 showed some curvature up to a phase ratio of several hundred but finally assumed a constant slope equivalent to a steady-state loss of 9 ppm:

Amine and Diluent	Range of Phase Ratios (a/o)	Cumulative Loss, % of initial amine	Average Loss Rate, ^a ppm
Amberlite LA-1 (comp'd. 193H) in kerosene	0-30	6	-
	30-200	18	23
	200-500	33	17
	500-1000	51	14
	1000-1500	62	8
S-24 (comp'd. 30B) in kerosene	0-50	2	-
	50-250	11	13
	250-750	27	11
	750-1000	33	9
	1000-1800	53	9

^aTo synthetic Marysvale liquor; analysis in Table 3.1.

With the exception of tri(iso-heptyl)amine, which was readily lost to the aqueous phase, all tertiary amines tested had a negligible or tolerably low loss. Tri(iso-octyl)amine gave the same type of loss curve as described above for Primene JM and Amberlite LA-1. The average loss to the first 1400 aqueous volumes contacted (excluding the initial loss of 4%) was ~11 ppm.

Amine and Diluent	Range of Phase Ratios (a/o)	Cumulative Loss, % of initial amine	Average Loss Rate, ppm
Tri(iso-octyl)	0-50	4	-
(comp'd. 239C)	50-400	22	16
in 97% kerosene--	400-800	36	11
3% tridecanol	800-1400	50	9

4.0 URANIUM EXTRACTION ISOTHERMS

Isotherms (Table 4.1) for the extraction of uranium from a synthetic leach liquor (synthetic Marysville liquor) were determined for 15 different amines (0.1 M in kerosene or kerosene-tridecanol diluent) that performed well in the preliminary extraction tests (Sec. 2.0). In general, extraction results were similar to those obtained previously⁶ with compounds of similar type and structure, the N-benzyl branched-alkyl secondaries showing the highest uranium extraction power and the tertiary amines the best selectivity for uranium over iron(III).

A new batch of S-24 amine gave extraction results approximately equivalent to those for a previous⁶ batch. This amine, and two other secondaries with branching of both alkyl chains close to the nitrogen, i.e., di(1-heptyloctyl) and di(1-nonyl-decyl) amines, showed high extraction coefficients (~300), good selectivity for uranium over iron(III), and saturation loadings of ~4.3 g of uranium per liter.

Extraction results for Amberlite LA-1 were similar to those obtained for a previous⁶ sample. Uranium extractions were slightly weaker and the selectivity slightly poorer than for the secondary amines described above. Addition of 3 vol % tridecanol to the kerosene diluent lowered extraction coefficients for both uranium and iron by a factor of 2-3. Amberlite LA-2 was more selective and loaded higher with uranium than Amberlite LA-1 but had much lower uranium extraction coefficients ($E_a^0 = 35$) and slower phase separation. Phase separation was faster (but still not rapid) and uranium coefficients slightly higher with alcohol added to the solvent.

As observed previously,³ modification of the kerosene diluent with alcohol greatly depressed the extraction of iron

Table 4.1. Uranium Extraction Isotherms from Synthetic Marysvale Liquor

Aqueous: 0.15-3.0 g of U, 5.0-5.8 g of Fe(III), 3.3 g of Al, 50 g of SO₄, 2.0 g of PO₄, and 1.7 g of F per liter, pH 0.90

Organic: 0.10 M amine in kerosene with indicated concentration of tridecanol (TDA)

Phase ratio, a/o: 2/1; contact time: 5 min

Amine	TDA Conc., vol %	Phase Sep'n Time, min	Uranium Conc., g/liter			Fe in Organic, g/liter	Uranium Extraction Coefficient (E _a ⁰)
			Head Liquor	Organic	Aqueous		
S-24 (Compound 30D)	0	<1	0.15	0.30	0.0009	-	330
		<1	0.30	0.61	0.002	0.18	310
		<1	0.60	-	0.004	0.13	~300
		<1	1.0	2.0	0.009	0.10	220
		<1	1.25	2.4	0.013	0.084	180
		<1	1.50	2.9	0.021	0.068	140
		<1	2.0	3.9	0.066	0.038	59
		<1	3.0	4.3	0.79	0.010	5
Di(1-heptyloctyl) (Compound 332A)	0	0.7	0.15	0.28	0.001	0.32	280
		0.8	0.30	0.56	0.002	0.27	280
		0.8	0.60	1.2	0.007	0.22	170
		0.7	1.0	2.0	0.014	0.17	140
		0.8	1.25	2.4	0.024	0.12	100
		1.0	1.50	3.0	0.038	0.10	80
		1.3	2.0	3.8	0.15	0.050	25
		1.4	3.0	4.3	0.85	0.027	5
Di(1-nonyldecyl) (Compound 341A)	0	0.6	0.15	0.29	0.001	0.23	290
		0.6	0.30	0.56	0.002	0.21	280
		0.5	0.60	1.2	0.006	0.18	200
		0.5	1.0	2.0	0.013	0.13	150
		0.4	1.25	2.4	0.024	0.099	100
		0.4	1.50	2.8	0.036	0.075	78
		0.5	2.0	3.8	0.16	0.029	24
		0.5	3.0	4.3	1.0	0.008	4

Table 4.1 (Cont'd.)

Amine	TDA Conc., vol %	Phase Sep'n Time, min	Uranium Conc., g/liter			Fe in Organic, g/liter	Uranium Extraction Coefficient (E_a^O)
			Liquor	Organic	Aqueous		
Amberlite LA-1 (Compound 193H)	0	~1	0.15	0.30	0.002	0.40	150
		~1	0.30	0.60	0.003	0.34	200
		~1	0.60	1.2	0.007	0.30	170
		~1	1.0	2.0	0.016	0.14	120
		~1	1.25	2.4	0.027	0.14	90
		~1	1.50	2.8	0.048	0.08	60
		~1	2.0	3.3	0.28	0.015	12
		~1	3.0	3.5	1.2	0.005	3
	3	1.0	0.15	0.29	0.003	0.19	100
		1.0	0.30	0.59	0.007	0.16	85
		1.0	0.60	1.2	0.017	0.13	70
		1.0	1.0	2.1	0.046	0.088	45
		1.0	1.25	2.4	0.080	0.061	30
		1.0	1.50	2.8	0.14	0.040	20
		1.0	2.0	3.3	0.39	0.048	8
		1.0	3.0	3.6	1.2	0.014	3
Amberlite LA-2 (Compound 325A)	0	>10	0.15	0.29	0.008	0.13	36
		>10	0.30	0.58	0.018	0.12	32
		4	0.60	1.2	0.045	0.10	27
		3.3	1.0	1.9	0.089	0.088	21
		2.0	1.25	2.4	0.13	0.068	18
		1.7	1.50	2.7	0.17	0.049	16
		1.4	2.0	3.4	0.32	0.037	11
		1.2	3.0	4.2	0.94	0.014	4
	3	1.2	0.15	0.28	0.007	0.077	40
		1.5	0.30	0.57	0.016	0.070	36
		1.5	0.60	1.1	0.040	0.060	28
		1.5	1.0	1.8	0.077	0.047	23

Table 4.1 (Cont'd.)

Amine	TDA Conc., vol %	Phase Sep'n Time, min	Uranium Conc., g/liter			Fe in Organic, g/liter	Uranium Extraction Coefficient (E_a^0)
			Head Liquor	Organic	Aqueous		
Amberlite LA-2 (Compound 325A)	3	1.5	1.25	2.2	0.11	0.037	20
		1.5	1.50	2.7	0.16	0.031	17
		1.5	2.0	3.3	0.32	0.024	10
		1.1	3.0	4.2	0.91	0.010	5
Amberlite LA-2 (Compound 325B)	3	1.5	0.15	0.30	0.009	0.084	33
		1.5	0.30	0.58	0.017	0.078	34
		1.7	0.60	1.1	0.044	0.064	25
		1.9	1.0	1.9	0.088	0.046	22
		1.7	1.25	2.4	0.13	0.045	18
		2.0	1.50	2.6	0.17	0.038	15
		1.9	2.0	3.7	0.33	0.036	11
		2.0	3.0	4.1	0.93	0.023	4
Di(tridecyl) (Compound 227A)	5	~1	0.15	0.34	0.002	0.40	170
		~1	0.30	0.60	0.005	0.33	120
		~1	0.60	1.1	0.011	0.32	100
		~1	1.0	1.9	0.026	0.28	75
		~1	1.25	2.3	0.042	0.14	55
		~1	1.50	2.8	0.067	0.090	40
		~1	2.0	3.3	0.20	0.026	16
		~1	3.0	3.9	0.90	0.008	4
Di(tridecyl) (Compound 227B)	0	1.3	0.15	0.31	0.004	1.1	80
		1.3	0.30	0.63	0.009	0.90	70
		1.5	0.60	1.2	0.020	0.77	60
		1.4	1.0	2.0	0.043	0.55	45
		2.0	1.25	2.3	0.073	0.43	30
		2.0	1.50	2.9	0.099	0.31	29
		2.0	2.0	3.7	0.23	0.14	16
		1.7	3.0	4.3	0.90	0.03	5

Table 4.1 (Cont'd.)

Amine	TDA Conc., vol %	Phase Sep'n Time, min	Uranium Conc., g/liter			Fe in Organic, g/liter	Uranium Extraction Coefficient (E _a ⁰)
			Head Liquor	Organic	Aqueous		
Di(tridecyl) (Compound 227B)	5	1.1	0.15	0.32	0.002	0.37	160
		0.8	0.30	0.63	0.005	0.33	130
		0.9	0.60	1.2	0.011	0.26	110
		0.9	1.0	2.0	0.023	0.18	85
		1.0	1.25	2.4	0.045	0.13	55
		1.0	1.50	2.9	0.072	0.091	40
		1.2	2.0	3.6	0.20	0.038	18
		1.1	3.0	4.4	0.98	0.015	4
N-Benzyl-(1-nonyldecyl) (Compound 330A)	3	2.1	0.15	0.30	0.0001	0.50	3000
		2.5	0.30	0.55	0.0003	0.45	1800
		2.5	0.60	1.2	0.0009	0.37	1300
		2.1	1.0	2.0	0.002	0.26	1000
		1.9	1.25	2.5	0.003	0.19	800
		1.6	1.50	2.9	0.004	0.12	700
		1.2	2.0	3.8	0.081	0.017	45
		1.2	3.0	3.9	1.1	0.010	4
N-Benzyl-(1-undecyl- dodecyl) (Compound 331A)	3	1.8	0.15	0.30	0.0002	0.57	1500
		1.8	0.30	0.56	0.0003	0.52	1900
		1.6	0.60	1.2	0.0005	0.42	2400
		1.4	1.0	2.1	0.002	0.27	1000
		1.4	1.25	2.5	0.002	0.22	1200
		1.3	1.50	2.9	0.005	0.16	600
		1.2	2.0	3.5	0.076	0.019	45
		1.1	3.0	4.0	1.1	0.012	4
Tri(iso-octyl) (Compound 239B)	3	<1	0.15	-	0.0009	0.011	~330
		<1	0.30	0.61	0.002	0.010	300
		<1	0.60	1.2	0.004	0.008	300
		<1	1.0	2.1	0.008	0.012	260

Table 4.1 (Cont'd.)

Amine	TDA Conc., vol %	Phase Sep'n Time, min	Uranium Conc., g/liter			Fe in Organic, g/liter	Uranium Extraction Coefficient (E_a^O)
			Head Liquor	Organic	Aqueous		
Tri(iso-octyl) (Compound 239B)	3	<1	1.25	2.6	0.012	0.006	220
		<1	1.50	3.1	0.019	0.005	160
		<1	2.0	4.1	0.042	0.004	100
		<1	3.0	5.4	0.42	0.002	13
Tri(iso-octyl) (Compound 239C)	3	1.0	0.15	0.25	0.0008	0.015	310
		1.0	0.30	0.60	0.002	0.013	300
		1.0	0.60	1.2	0.004	0.012	300
		1.0	1.0	2.0	0.008	—	250
		1.0	1.25	2.4	0.012	0.007	200
		1.0	1.50	2.8	0.020	0.006	140
		1.0	2.0	3.8	0.046	0.004	80
		1.0	3.0	5.0	0.41	<0.003	12
Alamine 336 (Compound 299A)	3	1.0	0.15	0.33	0.001	0.020	330
		1.0	0.30	0.66	0.002	0.019	330
		1.0	0.60	1.2	0.005	0.018	240
		1.0	1.0	1.9	0.011	0.016	170
		1.0	1.25	2.4	0.018	0.013	130
		1.0	1.50	2.8	0.025	0.011	110
		1.0	2.0	3.8	0.062	0.008	60
		1.0	3.0	4.8	0.50	0.003	10
Alamine 336 (Compound 299C)	3	0.5	0.15	0.30	0.0008	—	370
		0.5	0.30	0.63	0.002	—	310
		0.5	0.60	1.2	0.004	—	300
		0.5	1.50	2.9	0.027	—	110
		0.5	2.0	4.1	0.044	—	95
		0.5	3.0	5.5	0.39	—	14

Table 4.1 (Cont'd.)

Amine	TDA Conc., vol %	Phase Sep'n Time, min	Uranium Conc., g/liter			Fe in Organic, g/liter	Uranium Extraction Coefficient (E_a^O)
			Head Liquor	Organic	Aqueous		
ADM Mixed Tertiary (Compound 328A)	3	0.9	0.15	0.32	0.0006	0.016	530
		0.8	0.30	0.62	0.001	0.014	600
		0.8	0.60	1.2	0.003	0.014	400
		0.9	1.0	2.0	-	0.012	-
		0.5	1.25	2.5	-	0.009	-
		0.6	1.50	3.0	-	0.008	-
		0.6	2.0	3.6	0.048	0.008	75
		1.0	3.0	5.1	0.39	0.005	13
Dilauryl-n-butyl (Compound 101B)	3	1.8	0.15	0.29	0.0008	0.022	360
		0.8	0.30	0.60	0.002	0.019	300
		1.0	0.60	1.2	0.005	0.017	240
		1.2	1.0	2.1	0.025	0.015	85
		0.8	1.25	2.4	0.028	0.014	85
		1.1	1.50	2.8	0.040	0.011	70
		1.2	2.0	3.8	0.083	0.009	45
		1.3	3.0	5.1	0.38	0.006	13
Amberlite XE-204 (Compound 353A)	3	1.0	0.15	0.29	0.001	0.025	290
		1.0	0.30	0.62	0.002	0.021	310
		1.0	0.60	1.2	0.005	0.018	240
		1.0	1.0	2.0	0.013	0.012	150
		1.0	1.25	2.5	0.017	0.010	150
		1.0	1.50	3.0	0.024	0.008	120
		1.0	2.0	3.7	0.054	0.005	70
		1.0	3.0	5.1	0.38	<0.005	13

Table 4.1 (Cont'd.)

Amine	TDA Conc., vol %	Phase Sep'n Time, min	Uranium Conc., g/liter			Fe in Organic, g/liter	Uranium Extraction Coefficient (E_a^O)
			Head Liquor	Organic	Aqueous		
Amberlite XE-204 (Compound 353C)	3	1.1	0.15	0.28	0.001	0.026	280
		1.2	0.30	0.57	0.002	0.023	280
		1.8	0.60	1.2	0.006	0.022	200
		1.4	1.0	2.0	0.014	0.016	140
		1.1	1.25	2.5	0.021	0.014	120
		1.2	1.50	2.9	0.029	0.010	100
		1.4	2.0	4.0	0.061	0.009	65
		1.6	3.0	5.0	0.51	0.006	10
Trilauryl (Compound 86B)	3	-	0.15	0.32	0.001	0.028	320
		-	0.30	0.63	0.002	0.026	310
		-	0.60	1.2	0.005	0.023	240
		-	1.0	2.0	0.007	0.021	290
		-	1.25	2.5	0.014	0.017	180
		-	1.50	3.1	0.019	0.015	160
		-	2.0	4.0	0.042	0.011	95
		-	3.0	5.1	0.40	0.006	13
Trilauryl (Compound 86D)	3	6	0.15	0.31	0.0006	0.018	520
		6	0.30	0.60	0.001	0.017	600
		6	0.60	1.2	0.004	0.013	300
		8	1.0	1.9	0.007	0.011	270
		6	1.25	2.4	0.012	0.010	200
		6	1.50	2.9	0.014	0.010	200
		5	2.0	3.9	0.028	0.008	140
		4	3.0	5.0	0.35	0.004	14

Table 4.1 (Cont'd.)

Amine	TDA Conc., vol %	Phase Sep'n Time, min	Uranium Conc., g/liter			Fe in Organic, g/liter	Uranium Extraction Coefficient (E_a^0)
			Head Liquor	Organic	Aqueous		
Tris(tridecyl) (Compound 310A)	0	1.0	0.15	0.31	0.0004	0.017	770
		1.0	0.30	0.61	0.0009	0.015	680
		1.0	0.60	1.3	0.003	0.012	430
		1.0	1.0	2.0	0.005	0.009	400
		1.0	1.25	2.5	0.008	0.007	310
		1.0	1.50	3.1	0.012	0.005	260
		1.0	2.0	4.0	0.035	0.002	110
		1.0	3.0	4.9	0.52	0.001	9
Tris(tridecyl) (Compound 310A)	3	1.0	0.15	0.32	0.0008	0.006	400
		1.0	0.30	0.65	0.002	0.004	320
		1.0	0.60	1.2	0.004	0.003	300
		1.0	1.0	1.9	0.009	0.002	210
		1.0	1.25	2.3	0.018	0.002	130
		1.0	1.50	3.0	0.027	0.002	110
		1.0	2.0	3.9	0.084	0.001	45
		1.0	3.0	4.7	0.56	0.001	8

with di(tridecyl)amine. With 5 vol % tridecanol, the selectivity of this amine was only slightly poorer than that of Amberlite LA-1 in kerosene.

The two N-benzyl branched-alkyl secondary amines, N-benzyl(1-nonyldecyl) and N-benzyl(1-undecyldodecyl), gave extraction results similar to those obtained previously⁶ with N-benzyl-1-(3-ethylpentyl)-4-ethyloctylamine, uranium extraction coefficients at low loading being 2000-3000. Phase separation, which was extremely slow in kerosene diluent, was much faster but still not rapid with 3 vol % tridecanol added to the solvent.

All the tertiary amines, including Alamine 336, ADM mixed tertiary, butyldilauryl, Amberlite XE-204, trilauryl, tris(tridecyl), and two new batches of tri(iso-octyl)amine, showed the high uranium extraction power ($E_a^0 = 250-750$), excellent selectivity with respect to iron(III), and high uranium loadings (~5 g per liter) obtained previously⁶ with tri-n-octylamine and an earlier batch of tri(iso-octyl)amine. Phase separation was rapid (0.5-1.5 min) except for trilaurylamine, which was slow (4-8 min).

5.0 COMPATIBILITY WITH MOLYBDENUM

In application of the Amex process to certain molybdenum-containing liquors, amine-molybdenum precipitates (presumably heteropolymolybdates) sometimes formed,^{3,5} the tendency for precipitation being influenced by amine-diluent choice, molybdenum concentration, the presence of phosphate and vanadium(V), and the choice of stripping method. With respect to the latter, the tendency to precipitation was usually more pronounced in the chloride stripping method than in other stripping methods. Continued tests with some of the amines previously tested and with newer amines showed the branched chain secondary amines to have good molybdenum compatibility although, in some cases, amine precipitation occurred at high molybdenum loadings. With the exception of trilaurylamine, which had a high molybdenum tolerance, the tertiary amines showed relatively poor molybdenum compatibility.

5.1 Batch Extraction and Stripping Tests

A batch testing procedure similar to that described previously³ was used to compare the relative susceptibility of various amines to molybdenum precipitation. In these tests, 0.1 M amine solutions were loaded with uranium and molybdenum by contact with a series of synthetic leach liquors of varying molybdenum content. The uranium was stripped by three successive contacts with 1 M NaCl--0.05 M H₂SO₄ and the amine regenerated to the free base form and stripped of molybdenum by

contact with 0.3 M Na_2CO_3 . Each contact was for 5 min and the organic was allowed to stand 0.5 hr between contacts to determine whether or not precipitation had occurred. If precipitation was noticed at any point in the procedure, the test was stopped and a sample of the amine titrated for comparison with the head organic. The solids formed were of two general types: (1) amber to green, gummy precipitates which apparently contained amine since a significant amine loss was observed in almost every test in which they formed; and (2) white precipitates, much smaller in quantity (possibly only slow-breaking emulsions) which may not have contained amine, since amine losses where they formed were within the analytical uncertainty (2%) of the titration. Test results (Table 5.1) show a strong dependence on molybdenum loading of the solvent.

Amine S-24. Precipitation occurred during extraction at the highest molybdenum loading, ~3 g/liter, in unmodified kerosene but not in kerosene modified with 2 vol % tridecanol. Trace precipitation occurred in two of the chloride strip contacts and one regeneration contact but titrations indicated no significant amine loss.

Di(1-heptyloctyl)amine, N-benzyl-(1-nonyldecyl)amine, and N-benzyl-(1-undecyldodecyl)amine. These amines, which were tested only up to a molybdenum loading of ~1.2 g/liter showed no significant amine loss in any tests.

Di(tridecyl)amine. No precipitation occurred during extraction or stripping at molybdenum loadings up to 3 g/liter. Although trace precipitation occurred in regeneration, titrations showed no significant amine loss.

Amberlite LA-1. Precipitation occurred during extraction at the highest molybdenum loading, ~3 g/liter, but not during chloride stripping in any tests with loadings up to 1.2 g/liter. Trace precipitation occurred during regeneration, but titrations indicated no significant amine loss.

Amberlite LA-2. Precipitation occurred during extraction at molybdenum loadings approaching 3 g/liter in unmodified kerosene but not with 2 vol % tridecanol added to the solvent. Precipitation with some amine loss occurred in the regeneration step at molybdenum loadings of ~1.2 g/liter.

Tri(iso-octyl)amine. In kerosene-alcohol diluent, precipitation with significant amine loss occurred during chloride stripping at molybdenum loadings as low as ~0.5 g/liter and in the extraction stage at loadings of ~3 g/liter. The use of Esso heavy aromatic naphtha as diluent decreased the tendency to precipitate somewhat, but severe amine loss still occurred in chloride stripping at molybdenum loadings >1 g/liter.

Table 5.1. Molybdenum Compatibility Tests

Organic: 0.10 M amine in kerosene or kerosene-tridecanol (TDA) diluent

Liquors: Synthetic liquor (pH 1) containing, in grams per liter, 0.05-1.0 Mo, 1.0 U, 2 Fe(III), 2 Al, 1 V(IV), 50 SO₄ and 2 PO₄; aged 3-8 days before test

Chloride strip: 1.0 M NaCl--0.05 M H₂SO₄

Regeneration: ~0.3 M Na₂CO₃

Procedure: Organic contacted with liquor at organic/aqueous ratio of 1/3, stripped by three contacts with chloride solution each at organic/aqueous ratio of 2/1, and regenerated to free base form with carbonate at organic/aqueous ratio of 4/1; each contact for 5 min, with 0.5 hr stand between contacts for observation of precipitate formation

Organic		Mo Conc.,		Observations ^a					Amine Loss, % of Head
Amine	TDA vol %	g/liter		Ext'n.	Chloride Strip				
		Liquor	Extract		1st	2nd	3rd	Regen.	
S-24 (Compound 30D)	0	0.056	0.13	NP	NP	NP	NP	NP	<1
		0.11	0.30	NP	NP	NP	NP	NP	<1
		0.22	0.61	NP	NP	NP	TP	-	<1
		0.40	1.2	NP	NP	NP	TP	-	<1
		1.0	3 ^b	P	-	-	-	-	3
	2	0.056	0.13	NP	NP	NP	NP	NP	<1
		0.11	0.29	NP	NP	NP	NP	NP	<1
		0.22	0.60	NP	NP	NP	TP	-	<1
		0.40	1.2	NP	NP	NP	TP	NP	<1
		1.0	3.0	NP	NP	NP	NP	TP	2
Di(1-heptyloctyl) (Compound 332A)	0	0.1	0.26	NP	NP	NP	NP	NP	<1
		0.4	1.2	NP	NP	NP	NP	TP	<1

Table 5.1 (Cont'd.)

Organic		Mo Conc., g/liter		Observations ^a					Amine Loss, % of Head
Amine	TDA, vol %	Liquor	Extract	Ext'n.	Chloride Strip			Regen.	
					1st	2nd	3rd		
Di(tridecyl) (Compound 227A)	0	0.056	0.14	NP	NP	NP	NP	NP	<1
		0.11	0.30	NP	NP	NP	NP	NP	<1
		0.22	0.59	NP	NP	NP	NP	TP	<1
		0.40	1.2	NP	NP	NP	NP	TP	<1
		1.0	3.0	NP	NP	NP	NP	TP	<1
	2	0.056	0.14	NP	NP	NP	NP	NP	<1
		0.11	0.30	NP	NP	NP	NP	NP	<1
		0.22	0.59	NP	NP	NP	NP	TP	<1
		0.40	1.2	NP	NP	NP	NP	TP	<1
		1.0	3.0	NP	NP	NP	NP	TP	<1
Di(tridecyl) (Compound 227B)	0	0.11	0.27	NP	NP	NP	NP	NP	<1
		0.21	0.59	NP	NP	NP	NP	TP	<1
		0.40	1.2	NP	NP	NP	NP	TP	<1
		1.0	2.9	NP	NP	NP	NP	TP	<1
	5	0.11	0.24	NP	NP	NP	NP	NP	<1
		0.21	0.55	NP	NP	NP	NP	NP	<1
		0.40	0.97	NP	NP	NP	NP	TP	<1
		1.0	2.5	NP	NP	NP	NP	TP	<1
Amberlite LA-1 (Compound 193D)	0	0.056	0.16	NP	NP	NP	NP	TP	<1
		0.11	0.28	NP	NP	NP	NP	TP	1
		0.22	0.60	NP	NP	NP	NP	TP	<1
		0.40	1.0	NP	NP	NP	NP	TP	2
		1.0	3 ^b	P	-	-	-	-	3
Amberlite LA-1 (Compound 193D)	2	0.056	0.13	NP	NP	NP	NP	TP	<1
		0.11	0.26	NP	NP	NP	NP	TP	1
		0.22	0.56	NP	NP	NP	NP	TP	2
		0.40	1.2	NP	NP	NP	NP	TP	2
		1.0	3 ^b	P	-	-	-	-	6

Table 5.1 (Cont'd.)

Organic		Mo Conc.,		Observations ^a					Amine Loss, % of Head	
Amine	TDA, vol %	g/liter		Ext'n.	Chloride Strip			Regen.		
		Liquor	Extract		1st	2nd	3rd			
Amberlite LA-2 (Compound 325A)	0	0.11	0.25	NP	NP	NP	NP	NP	<1	
		0.21	0.54	NP	NP	NP	NP	NP	<1	
		0.40	1.2	NP	NP	NP	NP	P	3	
		1.0	3 ^b	P	-	-	-	-	3	
	3	0.11	0.24	NP	NP	NP	NP	NP	<1	
		0.21	0.55	NP	NP	NP	NP	NP	<1	
		0.40	1.1	NP	NP	NP	NP	NP	<1	
		1.0	2.7	NP	NP	NP	NP	P	2	
	3	0.1	0.25	NP	NP	NP	NP	NP	1	
		0.4	1.2	NP	NP	NP	NP	NP	<1	
	N-Benzyl-(1-nonyl- decyl) (Compound 330A)	3	0.1	0.24	NP	NP	NP	NP	NP	<1
			0.4	1.1	NP	NP	NP	NP	NP	<1
N-Benzyl-(1-undecyl- dodecyl) (Compound 331A)	3	0.1	0.24	NP	NP	NP	NP	NP	<1	
		0.4	1.1	NP	NP	NP	NP	NP	<1	
Tri(iso-octyl) (Compound 239B)	5	0.056	0.12	NP	NP	NP	NP	TP	<1	
		0.11	0.29	NP	NP	NP	NP	TP	1	
		0.22	0.56	NP	NP	NP	P	-	3	
		0.40	0.87	NP	P	-	-	-	7	
		1.0	3 ^b	P	-	-	-	-	10	
	8	0.056	0.09	NP	NP	NP	NP	NP	<1	
		0.11	0.24	NP	NP	NP	NP	TP	<1	
		0.22	0.46	NP	NP	NP	P	-	4	
		0.40	0.71	NP	P	-	-	-	8	
		1.0	3 ^b	P	-	-	-	-	10	

Table 5.1 (Cont'd.)

Organic Amine	TDA, vol %	Mo Conc., g/liter		Ext'n.	Observations ^a				Amine Loss, % of Head
		Liquor	Extract		Chloride 1st	Strip 2nd	3rd	Regen.	
Tri(iso-octyl) (Compound 239B)	0 ^c	0.056	0.16	NP	NP	NP	NP	NP	<1
		0.11	0.31	NP	NP	NP	NP	NP	<1
		0.22	0.62	NP	NP	NP	NP	NP	2
		0.40	1.2	NP	NP	P	-	-	7
		1.0	3.0	NP	P	-	-	-	20
	2 ^c	0.056	0.15	NP	NP	NP	NP	NP	<1
		0.11	0.31	NP	NP	NP	NP	NP	<1
		0.22	0.59	NP	NP	NP	NP	NP	<1
		0.40	1.2	NP	NP	P	-	-	6
		1.0	3.1	NP	P	-	-	-	20
Alamine 336 (Compound 299E)	3	0.11	0.30	NP	NP	NP	NP	P	2
		0.44	1.2	NP	TP	P	-	-	4
		1.1	3.3	NP	P	-	-	-	14
ADM Mixed Tertiary (Compound 328A)	3	0.1	0.27	NP	NP	NP	NP	TP	2
		0.4	1.2	NP	NP	NP	P	-	<1
Butyldilauryl (Compound 101B)	3	0.1	0.27	NP	NP	NP	NP	TP	2
		0.4	1.0	NP	NP	NP	NP	P	5
Amberlite XE-204 (Compound 353C)	3	0.11	0.30	NP	NP	NP	NP	P	1
		0.44	1.2	NP	NP	NP	P	-	3
		1.1	3.3	NP	P	-	-	-	12
Trilauryl (Compound 86B)	3	0.11	0.29	NP	NP	NP	TP	NP	<1
		0.41	1.0	NP	NP	NP	TP	NP	<1
	5	0.11	0.30	NP	NP	NP	TP	NP	1
		0.41	1.1	NP	NP	NP	TP	NP	<1

Table 5.1 (Cont'd.)

Organic		Mo Conc.,		Observations ^a					Amine Loss, % of Head	
Amine	TDA, vol %	g/liter		Ext'n.	Chloride Strip					
		Liquor	Extract		1st	2nd	3rd	Regen.		
Trilauryl (Compound 86D)	3	0.11	0.25	NP	NP	NP	NP	NP	<1	
		0.21	0.45	NP	NP	NP	NP	NP	<1	
		0.40	1.0	NP	NP	NP	NP	TP	<1	
		1.0	2.6	NP	NP	NP	NP	TP	<1	
	5	0.11	0.25	NP	NP	NP	NP	NP	<1	
		0.21	0.46	NP	NP	NP	NP	NP	<1	
		0.40	0.88	NP	NP	NP	TP	NP	<1	
		1.0	2.3	NP	NP	NP	TP	TP	<1	
	0	0.1	0.31	NP	NP	NP	TP	NP	2	
		0.4	1.2	NP	TP	P	-	-	4	
		3	0.1	0.3	NP	NP	NP	TP	NP	1
			0.4	1.2	NP	TP	P	-	-	4

^aNP = no precipitate; TP = trace of white material present, possibly precipitate;
P = amber to green, gummy precipitate.

^bMolybdenum concentration expected if precipitation had not occurred during
extraction.

^cDiluent = Esso Heavy Aromatic Naphtha, scrubbed before use with sulfuric acid and
sodium carbonate solutions.

Alamine 336. Precipitation and amine loss occurred in the regeneration step at a molybdenum loading of ~0.3 g/liter, in chloride stripping at 1.2 g/liter, and in extraction at ~3 g/liter.

ADM Mixed Tertiary. Precipitation occurred in regeneration at a molybdenum loading of ~0.3 g/liter and in chloride stripping at 1.2 g/liter, but indicated amine losses were not significant.

Butyldilaurylamine. Precipitation and significant amine loss occurred in the regeneration step at a molybdenum loading of ~1 g/liter.

Amberlite XE-204. Precipitation occurred in the regeneration step at a molybdenum loading of 0.3 g/liter and in chloride stripping at 1.2 g/liter.

Trilaurylamine. No precipitation occurred in extraction or chloride stripping even up to molybdenum loadings of ~2.5 g/liter. Although trace precipitation was observed in the regeneration step, titrations indicated no amine loss.

Tris(tridecyl)amine. In chloride stripping, trace precipitation occurred at a molybdenum loading of 0.3 g/liter and precipitation with significant amine loss at 1.2 g/liter.

5.2 Continuous Countercurrent Tests

In continuous tests with Amine S-24 and Amberlite LA-1, no precipitation occurred in demonstration of the Amex process for recovering uranium and molybdenum from a synthetic leach liquor similar to those obtained by leaching Dakota lignite. The liquor (pH 1) contained, in grams per liter: 0.3 U, 0.3 Mo, 15 Fe(III), 5 Al, 10 PO₄, and 160 SO₄. Uranium and molybdenum were co-extracted with 0.1 M amine in kerosene, the loading being approximately 2 g of uranium and 2 g of molybdenum per liter. The uranium was selectively stripped in four stages with 0.9 M NaCl--0.1 M HCl, and then the molybdenum was stripped in three stages with 0.95 M Na₂CO₃. The flow ratios of head liquor/organic/chloride strip/carbonate strip were 30/4/1/1.

In general, the compatibility studies described above emphasize that caution must be observed in applying amines to solvent extraction recovery of uranium from liquors containing appreciable amounts of molybdenum. It should be emphasized that the tests are useful only for comparing the relative compatibility of different amines with molybdenum liquors under these particular test conditions, rather than for defining the molybdenum loading of the solvent that can be tolerated in process practice. The latter is dependent on liquor composition, e.g., concentration of phosphate, etc., and for maximum safety, the amine should be checked in a continuous circuit with the

actual liquor that is to be processed. Results of the above and other tests suggest that process difficulties can be avoided by appropriate choice of amine and stripping flow-sheet. Other workers,⁷ in tests with Alamine 336, with conditions under which copious amine-molybdenum precipitation would be expected, prevented precipitation by scrubbing the solvent with a dilute solution of sodium sulfide or sodium hydrogen sulfide prior to chloride stripping.

6.0 REFERENCES

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2. J. G. Moore, K. B. Brown, and C. F. Coleman, "Further Studies of Amines as Extractants for Uranium from Acid Sulfate Solutions," ORNL-1922 (Aug. 9, 1955).
3. D. J. Crouse et al., "Progress Report on Uranium Extraction with Organonitrogen Compounds," ORNL-2099 (June 15, 1956).
4. K. B. Brown et al., "Progress Report on Raw Materials for February, 1957," ORNL-2269 (May 31, 1957).
5. B. S. Weaver and D. E. Horner, "Distribution Behavior of Neptunium and Plutonium between Acid Solutions and Some Organic Extractants," J. Chem. Eng. Data, 5:260 (1960).
6. D. J. Crouse and K. B. Brown, "Amine Extraction Processes for Uranium Recovery from Sulfate Liquors," ORNL-1959 (Nov. 18, 1955).
7. J. E. House, General Mills, letter to D. J. Crouse, Nov. 25, 1958.

7.0 APPENDIX

Description of Organonitrogen Compounds

Available information on the structure, source, and purity of the organonitrogen compounds discussed in this report is listed in Table 7.1. The procedures (nonaqueous titrations) used for determining the equivalent weight, and the primary, secondary, and tertiary amine content of the samples are described in ORNL-1922.²

In the preliminary uranium extraction tests (Sec. 2.0) and the solubility loss measurements (Sec. 3.0), the amine samples were simply diluted with diluent prior to use. However, in the tests described in Secs. 4.0 and 5.0, the amine, dissolved in diluent, was scrubbed with dilute sulfuric acid and sodium carbonate prior to use in order to remove soluble impurities that could affect test results.

Table 7.1 Description of Compounds

Compound	Batch No.	Source ^a	Equiv. Wt.		Composition, %			Formula
			Theo.	Found	RNH ₂	R ₂ NH	R ₃ N	
Primary Amines								
21F81	120C	C	255	255	>99	-	-	$\begin{array}{c} \text{CH}_2\text{CH}_3 \\ \\ \text{CH}_3\text{CH}_2\text{CH}_2\text{CH}_2\text{CHCH}_2\text{CH}_2\text{CH}-\text{NH} \\ \\ \text{CH}_2\text{CH}_2\text{CH}(\text{CH}_2\text{CH}_3)_2 \end{array}$
1-Heptyloctyl	270A	A	227	238	99	<1	<1	(C ₇ H ₁₅) ₂ CH-NH ₂
1-Nonyldecyl	336A	A	284	291	-	-	-	(C ₉ H ₁₉) ₂ CH-NH ₂
	336B	A	284	286	>99	-	-	
1-Undecyl- dodecyl	272A	A	340	368	97	2	<1	(C ₁₁ H ₂₃) ₂ CH-NH ₂
	272B	A	340	350	98	<1	1	
Primene JM-T	6E	R	-	343	>99	-	-	R,R',R''-C-NH ₂ , where the R's contain 18-22 carbon atoms
Armeen O	275A	A	287	288	-	-	-	Red oil amine derived from oleic acid
Armeen OD	276A	A	265	266	-	-	-	Distilled Armeen O
Alkyl ether amine No. 1	304A	ADM	240-260 ^b	285	-	-	-	R-O-(CH ₂) ₃ -NH ₂ , R probably C ₁₂
Alkyl ether amine No. 3	305A	ADM	310-330 ^b	384	-	-	-	R-O-(CH ₂) ₃ -NH ₂ , R probably C ₁₈
Alkyl ether amine No. 4	306A	ADM	310-330 ^b	369	-	-	-	R-O-(CH ₂) ₃ -NH ₂ , R probably unsaturated C ₁₈
Secondary Amines								
S-24	30D	C	354	364	<1	98	2	$\left[(\text{CH}_3)_2\text{CHCH}_2\underset{\text{CH}_3}{\text{CHCH}_2}\underset{\text{CH}_2\text{CH}(\text{CH}_3)_2}{\text{CH}} \right]_2-\text{NH}$

Table 7.1 (Cont'd.)

Compound	Batch No.	Source ^a	Equiv. Wt.		Composition, %			Formula
			Theo.	Found	RNH ₂	R ₂ NH	R ₃ N	
Di(tridecyl)	227B	C	382	405	4	94	<2	(C ₁₃ H ₂₇) ₂ NH, alkyl group derived from tetra propylene
Amberlite LA-1 (9D-178)	193G	R	-	348	-	-	-	(CH ₃) ₂
	193H	R	-	370	7	75	18	(CH ₃) ₃ CCH ₂ CCH ₂ CH:CHCH ₂ -NH-CRR'R'', R's containing 12-14 carbon atoms
Amberlite LA-2	325A	R	-	366	1	97	2	C ₁₂ H ₂₅ -NH-CRR'R'', R's containing 12-14 carbon atoms
	325B	R	-	365	2	91-94	4-7	
Di(1-heptyl-octyl)	332A	A	438	436	6	91	2	(C ₇ H ₁₅) ₂ CH-NH-CH(C ₇ H ₁₅) ₂
Di(1-nonyl-decyl)	341A	A	549	547	3	95	1	(C ₉ H ₁₉) ₂ CH-NH-CH(C ₉ H ₁₉) ₂
N-(1-nonyl-decyl)dodecyl	309A	A	452	450	6	92	2	(C ₉ H ₁₉) ₂ CH-NH-C ₁₂ H ₂₅
N-(1-undecyl-dodecyl)-dodecyl	308A	A	508	513	17	81	2	(C ₁₁ H ₂₃) ₂ CH-NH-C ₁₂ H ₂₅
Benzyl-isopropyl	307A	S	149	155	-	-	-	C ₆ H ₅ -CH ₂ -NH-CH(CH ₃) ₂
N-Benzyl-1-isobutyl-3,5-dimethylhexyl	244B	CU	275	277	5	92	3	$ \begin{array}{c} \text{CH}_3 \\ \\ \text{C}_6\text{H}_5-\text{CH}_2-\text{NH}-\text{CHCH}_2\text{CHCH}_2\text{CH}(\text{CH}_3)_2 \\ \\ \text{CH}_2\text{CH}(\text{CH}_3)_2 \end{array} $
N-Benzyl-1-(3-ethylpentyl)-4-ethyloctyl	228B	C	347	357	4	95	<1	$ \begin{array}{c} \text{CH}_2\text{CH}_3 \\ \\ \text{C}_6\text{H}_5-\text{CH}_2-\text{NH}-\text{CHCH}_2\text{CH}_2\text{CHCH}_2\text{CH}_2\text{CH}_2\text{CH}_3 \\ \\ \text{CH}_2\text{CH}_2\text{CH}(\text{CH}_2\text{CH}_3)_2 \end{array} $

Table 7.1 (Cont'd.)

Compound	Batch No.	Source ^a	Equiv. Wt.		Composition, %			Formula
			Theo.	Found	RNH ₂	R ₂ NH	R ₃ N	
N-Benzyl (1-nonyl-decyl)	330A 330B	A A	374 374	379 388	2 4	98 93	<1 2	C ₆ H ₅ -CH ₂ -NH-CH(C ₉ H ₁₉) ₂
N-Benzyl (1-undecyldodecyl)	331A	A	430	435	2	98	<1	C ₆ H ₅ -CH ₂ -NH-CH(C ₁₁ H ₂₃) ₂
N-Benzyl octadecyl	327A	A	360	379	5	95	0	C ₆ H ₅ -CH ₂ -NH-C ₁₈ H ₃₇
N-Benzyl (1-methyloctadecyl)	329A	A	374	351	3	95	2	C ₆ H ₅ -CH ₂ -NH-CH(CH ₃)-C ₁₇ H ₃₅
4-RKB-21, N-(cyclohexylmethyl)-1-isobutyl-3,5-dimethylhexyl	297A	C	282	281	2	96	1	$\text{CH}_2(\text{CH}_2)_4\text{CHCH}_2\text{-NH-CHCH}_2\overset{\text{CH}_3}{\underset{\text{CH}_2\text{CH}(\text{CH}_3)_2}{\text{CHCH}_2\text{CH}(\text{CH}_3)_2}}$
4-RKB-20, N-(1-methylcyclohexylmethyl)-1-isobutyl-3,5-dimethylhexyl	296A	C	296	295	2	97	<1	$\text{CH}_2(\text{CH}_2)_4\overset{\text{CH}_3}{\underset{\text{CH}_3}{\text{C}}}\text{CH}_2\text{-NH-CHCH}_2\overset{\text{CH}_3}{\underset{\text{CH}_2\text{CH}(\text{CH}_3)_2}{\text{CHCH}_2\text{CH}(\text{CH}_3)_2}}$
4-RKB-16, N-(2,4,6-trimethylcyclohexylmethyl)-1-isobutyl-3,5-dimethylhexyl	293A	C	324	326	4	94	1	$\text{CHCH}_2\overset{\text{CH}_3}{\underset{\text{CH}_3}{\text{CH}}}\text{CH}_2\overset{\text{CH}_3}{\underset{\text{CH}_3}{\text{CH}}}\text{CH}_2\text{-NH-CHCH}_2\overset{\text{CH}_3}{\underset{\text{CH}_2\text{CH}(\text{CH}_3)_2}{\text{CHCH}_2\text{CH}(\text{CH}_3)_2}}$
4-RKB-19, N-cyclohexylmethyl-1-(3-ethylpentyl)-4-ethyloctyl	295A	C	352	349	6	94	<1	$\text{CH}_2(\text{CH}_2)_4\text{CHCH}_2\text{-NH-CHCH}_2\overset{\text{CH}_2\text{CH}_3}{\underset{\text{CH}_2\text{CH}_2\text{CH}(\text{CH}_2\text{CH}_3)_2}{\text{CH}_2\text{CH}_2\text{CH}_2\text{CH}_2\text{CH}_3}}$

Table 7.1 (Cont'd.)

Compound	Batch No.	Source ^a	Equiv. Wt.		Composition, %			Formula
			Theo.	Found	RNH ₂	R ₂ NH	R ₃ N	
4-RKB-18, N-(1-methylcyclohexylmethyl)-1-(3-ethylpentyl)-4-ethyloctyl	294A	C	366	363	2	97	1	$\overbrace{\text{CH}_2(\text{CH}_2)_4}^{\text{CH}_2\text{CH}_3} \underset{\text{CH}_3}{\text{C}}\text{CH}_2-\text{NH}-\underset{\text{CH}_2\text{CH}_2\text{CH}(\text{CH}_2\text{CH}_3)_2}{\text{CH}}\text{CH}_2\text{CH}_2\underset{\text{CH}_2\text{CH}_3}{\text{CH}}\text{CH}_2\text{CH}_2\text{CH}_2\text{CH}_3$
4-RKB-15, N-(2,4,6-trimethylcyclohexylmethyl)-1-(3-ethylpentyl)-4-ethyloctyl	292A	C	408	392	4	95	<1	$\overbrace{\text{CHCH}_2\text{CHCH}_2\text{CHCH}_2}^{\text{CH}_2\text{CH}_3} \underset{\text{CH}_3}{\text{CH}}\text{CH}_2-\text{NH}-\underset{\text{CH}_2\text{CH}_2\text{CH}(\text{CH}_2\text{CH}_3)_2}{\text{CH}}\text{CH}_2\text{CH}_2\underset{\text{CH}_2\text{CH}_3}{\text{CH}}\text{CH}_2\text{CH}_2\text{CH}_2\text{CH}_3$
Di(2-propyl-4-methylpentyl)	311A	EA	270	275	2	97	<1	$\left[\underset{\text{CH}_2\text{CH}_2\text{CH}_3}{\underset{\text{CH}_3}{(\text{CH}_3)_2\text{CHCH}_2\text{CHCH}_2}} \right]_2 - \text{NH}$
Mixed Secondary Amine	298A	GM	-	253	4	95	1	Mixed n-octyl and n-decyl
N-(p-sec-amylbenzyl)-1-isobutyl-3,5-dimethylhexyl	251A	C	346	527	-	-	-	$\text{CH}_3\text{CH}_2\text{CH}_2\underset{\text{CH}_3}{\text{CH}}-\text{C}_6\text{H}_4-\text{CH}_2-\text{NH}-\underset{\text{CH}_2\text{CH}(\text{CH}_3)_2}{\underset{\text{CH}_3}{\text{CH}}}\text{CH}_2\text{CH}(\text{CH}_3)_2$
N-(2-naphthylmethyl)-1-isobutyl-3,5-dimethylhexyl	252A	C	328	340	-	-	-	$\text{C}_{10}\text{H}_7-\text{CH}_2-\text{NH}-\underset{\text{CH}_2\text{CH}(\text{CH}_3)_2}{\underset{\text{CH}_3}{\text{CH}}}\text{CH}_2\text{CH}(\text{CH}_3)_2$

Table 7.1 (Cont'd.)

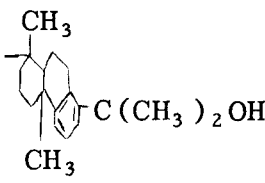
Compound	Batch No.	Source ^a	Equiv. Wt.		Composition, %			Formula
			Theo.	Found	RNH ₂	R ₂ NH	R ₃ N	
Rosin Amine X8523-83-7	277A	H	-	571	-	-	-	Derivatives of dehydroabietylamine; structures unknown
Rosin Amine HX-N-RA-60A	333A	H	-	390	-	-	-	
Rosin Amine HX-N-RA-61A	334A	H	-	402	-	-	-	
Polyrad 0100	282A	H	360 ^b	363	-	-	-	HOCH ₂ CH ₂ -NH-CH ₂ - 
<u>Tertiary Amines</u>								
Di(2-ethylhexyl)-n-hexyl	195A	C	326	319	1	10	89	$\left(\text{CH}_3\text{CH}_2\text{CH}_2\text{CH}_2\underset{\text{CH}_2\text{CH}_3}{\text{CH}}\text{CH}_2 \right)_2\text{-N-C}_6\text{H}_{13}$
Tri(2-ethylhexyl)	87A	ORNL	354	353	1	5	94	$\left(\text{CH}_3\text{CH}_2\text{CH}_2\text{CH}_2\underset{\text{CH}_2\text{CH}_3}{\text{CH}}\text{CH}_2 \right)_3\text{-N}$
	87B	EA	354	355	<1	27	73	
Tri(iso-heptyl)	326A	G	312	314	2	2	96	(C ₇ H ₁₅) ₃ N, alkyl groups branched
Tri-n-octyl	125H	C	354	350	3	1	96	(C ₈ H ₁₇) ₃ N
Tri(iso-octyl)	239A	C	354	357	5	2	93	(C ₈ H ₁₇) ₃ N, mixed 8-carbon alkyl groups branched
	239B	G	354	353	2	1	96	
	239C	C	354	358	1	9	89	
Alamine 336	299D	GM	-	416	<1	1	98	Mixed n-octyl and n-decyl
	299E	GM	-	411	<1	1	98	
	299F	GM	-	410	-	-	-	
ADM Mixed Tertiary	328A	ADM	-	464	<1	1-4	95-99	A mixture of dilauryl octyl and dilauryl decyl amines

Table 7.1 (Cont'd.)

Compound	Batch No.	Source ^a	Equiv. Wt.		Composition, %			Formula
			Theo.	Found	RNH ₂	R ₂ NH	R ₃ N	
Tri-n-decyl	84D	B	438	477	-	-	-	(C ₁₀ H ₂₁) ₃ N
Dilauryl-n-butyl	101A	ORNL	410	418	1	5	93	(C ₁₂ H ₂₅) ₂ -N-(C ₄ H ₉)
	101B	ADM	410	424	<1	2-5	95-98	
XE-204 (didodecenyl-n-butyl)	253A	R	406	413	2	1	97	[(CH ₃) ₃ CCH ₂ C(CH ₃) ₂ CH ₂ CH=CHCH ₂] ₂ -N-C ₄ H ₉
	253B	R	406	433	2	7	91	
	253C	R	406	439	-	-	-	
Trilauryl	86B	ADM	522	542	2	9	89	(C ₁₂ H ₂₅) ₃ N
	86C	A	522	600	7	3	89	
	86D	ADM	522	560	4	3	92	
	86D ^c	ADM	522	570	4	5	91	
Tris(tridecyl)	310A	C	564	566	2	1	97	(C ₁₃ H ₂₇) ₃ N with the alkyl group derived from tetrapropylene
Propomeen 212/11, n=1	313A	A	412	408	3	9	87	(C ₁₂ H ₂₅) ₂ -N-(CH ₂ CHO) _n -H CH ₃
Propomeen 212/12, n=2	319A	A	470	503	3	2	95	
Propomeen 212/13, n=3	320A	A	528	508	3	2	95	
Propomeen 212/14, n=4	322A	A	586	637	-	-	-	
Propomeen 212/20, n=10	323A	A	934	950	-	-	-	
Propomeen 212/50, n=40	324A	A	2677	2555	-	-	-	
RD-2805B, x+y = 2	300A	A	-	341	-	-	-	RN $\begin{cases} \text{[CH}_2\text{CH(CH}_3\text{)O]}_x\text{H} \\ \text{[CH}_2\text{CH(CH}_3\text{)O]}_y\text{H} \end{cases}$
RD-2806B, x+y = 5	301A	A	-	368	-	-	-	
RD-2807B, x+y = 8.5	302A	A	-	635	-	-	-	
RD-2808B, x+y = 15	303A	A	-	1060	-	-	-	
Armeen M-2C	248A	A	-	427	6	0	95	R ₂ -N-CH ₃ , R derived from coconut oil
Armeen M-2S	249A	A	-	582	3	0	98	R ₂ -N-CH ₃ , R derived from soya

Table 7.1 (Cont'd.)

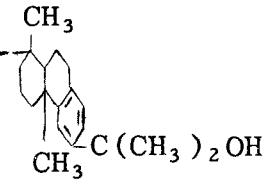
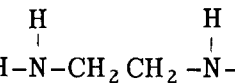
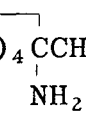
Compound	Batch No.	Source ^a	Equiv. Wt. Composition, %		Composition, %			Formula
			Theo.	Found	RNH ₂	R ₂ NH	R ₃ N	
Armeen M-2HT	250A	A	-	550	4	1	95	R ₂ -N-CH ₃ , R derived from hydrogenated tallow
Polyrad 0200	283A	H	402 ^b	419	-	-	-	(HOCH ₂ CH ₂) ₂ -N-CH ₂ - 
Miscellaneous Organonitrogen Compounds								
Arquad 18	291A	A	348	343	-	-	-	[C ₁₈ H ₃₇ -N-(CH ₃) ₃] ⁺ Cl ⁻
Quaternary B-104 (Didodecenyl- dimethyl- ammonium chloride)	247A	R	414	647	-	-	-	[{(CH ₃) ₃ CCH ₂ C(CH ₃) ₂ CH ₂ CH=CHCH ₂] ₂ N(CH ₃) ₂] ⁺ Cl ⁻
Arquad 2C	105B	A	-	429	-	-	-	[R-N(CH ₃) ₂ -R] ⁺ Cl ⁻ , R derived from coconut oil
Arquad 2T	290A	A	-	647	-	-	-	[R-N(CH ₃) ₂ -R] ⁺ Cl ⁻ , R derived from tallow
N,N'-bis-(1- heptyloctyl)- ethylene- diamine	271A	A	240	192	-	-	-	(C ₇ H ₁₅) ₂ CH-  -N-CH(C ₇ H ₁₅) ₂
Diamine 26	281A	GM	-	189	-	-	-	RNH-CH ₂ CH ₂ CH ₂ -NH ₂ , R derived from tallow
Di(1-amino- cyclohexyl- methyl)amine	312A	D	239	259	-	-	-	[CH ₂ (CH ₂) ₄  CCH ₂] ₂ -NH

Table 7.1 (Cont'd.)

Compound	Batch No.	Source ^a	Equiv. Wt.		Composition, %			Formula
			Theo.	Found	RNH ₂	R ₂ NH	R ₃ N	
Fatchemco DM-O	256A	U	366	349	-	-	-	$\text{C}_{17}\text{H}_{32}-\overset{\text{O}}{\underset{\text{H}}{\underset{ }{\text{C}}}}-\text{N}-\text{CH}_2\text{CH}_2\text{CH}_2-\text{N}(\text{CH}_3)_2$
Deriphat 150A	278A	GM	-	144	-	-	-	$\text{C}_{12}\text{H}_{25}-\text{NH}-\text{CH}_2\text{CH}_2-\overset{\text{O}}{\underset{ }{\text{C}}}-\text{ONa}$
Deriphat 160	280A	GM	-	132	-	-	-	"solubilized version of Deriphat 150A"
Deriphat 157	279A	GM	-	176	-	-	-	$\text{R}-\text{NH}-\text{CH}_2\text{CH}_2-\overset{\text{O}}{\underset{ }{\text{C}}}-\text{ONa}, \text{ R derived from tallow}$
Arneel S	269A	A	-	d	-	-	-	R-C≡N, R derived from soya fatty acid
Alkylaniline C-5	254A	M	-	179	-	-	-	R-C ₆ H ₄ -NH ₂ , R ranges from C ₃ H ₇ to C ₉ H ₁₉ , and averages C ₅ H ₁₁
Suconox 12	262A	S	291 ^b	-	-	-	-	$\text{H}-\text{N}-\overset{\text{O}}{\underset{ }{\text{C}}}-\text{R}$  R primarily C₁₁H₂₃
Suconox 18	263A	S	375 ^b	-	-	-	-	$\text{H}-\text{N}-\overset{\text{O}}{\underset{ }{\text{C}}}-\text{R}$  R approximately equal parts of C₁₅H₃₁ and C₁₇H₃₅
Ethoquad C/25	314A	A	-	d	-	-	-	$\left[\begin{array}{c} \text{CH}_3 \qquad \qquad \text{CH}_3 \\ \qquad \qquad \qquad \\ \text{R}-\text{N}-\text{CH}_2\text{CH}_2\text{CH}_2-\text{N}-(\text{CH}_2\text{CH}_2\text{O})_z\text{H} \\ \qquad \qquad \qquad \\ (\text{CH}_2\text{CH}_2\text{O})_x\text{H} \quad (\text{CH}_2\text{CH}_2\text{O})_y\text{H} \end{array} \right]^{++} \text{Cl}_2^-$ x+y+z = 15

Table 7.1 (Cont'd.)

Compound	Batch No.	Source ^a	Equiv. Wt.		Composition, %			Formula
			Theo.	Found	RNH ₂	R ₂ NH	R ₃ N	
O.C. No. 30	315A	A	-	d	-	-	-	Arquad C - urea complex
Emery 578-41R	287A	E	-	287	-	-	-	Acetate salts of amine
Emery 578-42R	288A	E	-	429	-	-	-	derivatives -
Emery 578-49R	289A	E	-	420	-	-	-	structures unknown
Duomeen T salt of polydodecylbenzene-sulfonic acid	284A	L	-	d	-	-	-	Structure unknown
Aminopolybutene	285A	L	-	d	-	-	-	Structure unknown
Imidazoline from fatty acid mixture and triethylene tetramine	286A	L	-	156	-	-	-	Structure unknown

- ^aA = Armour Chemical Division, Chicago
 ADM = Archer-Daniels-Midland Co., Minneapolis, Minn.
 B = Bios Laboratories, New York
 C = Union Carbide Chemicals Co., New York
 CU = Columbia University, New York
 D = E. I. duPont de Nemours and Company, Wilmington
 E = Emery Industries, Inc., Cincinnati
 EA = Eastman Chemical Products, Kingsport, Tenn.
 GM = General Mills, Inc., Kankakee, Ill.
 G = Gulf Oil Corp., Pittsburgh
 H = Hercules Powder Co., Wilmington
 L = The Lubrizol Corp., Cleveland
 M = Monsanto Chemical Co., St. Louis
 ORNL = Oak Ridge National Laboratory
 R = Rohm and Haas Co., Philadelphia
 S = Sumner Chemical Co., Inc., New York
 U = Universal Chemical Corp., Lonsdale, R. I.

^bEquivalent weight (or molecular weight of neutral compound) supplied by vendor.

^cRefrigerated and centrifuged - no solids present.

^dEquivalent weight could not be determined.

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